

In the Claims.

1. (Cancelled.)

2. (Original.) A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

conditioning the lipid feedstock in a conditioning reactor, wherein the lipid feedstock is heated, mixed and filtered to produce a conditioned lipid feedstock;

reacting the free fatty acid in the conditioned lipid feedstock with a purified glycerin product in a glycerolysis reactor, wherein the free fatty acid in the feedstock is mixed and continuously reacted with the purified glycerin product in the absence of a catalyst at an appropriate temperature and pressure in a glycerolysis reaction to produce a glycerolysis reactor effluent stream that contains a glyceride, the purified glycerin product being continuously added to the glycerolysis reactor at a rate that is greater than the stoichiometric amount of glycerin required for the glycerolysis reaction, water being continuously removed from the glycerolysis reactor as a vapor;

reacting the glyceride contained in the glycerolysis effluent stream with a purified alcohol product comprising an alcohol in a transesterification reactor, wherein the glyceride is mixed with said purified alcohol product and continuously reacted with the alcohol at an appropriate temperature and pressure in an alkali catalyzed transesterification reaction to produce a transesterification reactor effluent stream that contains a fatty acid alkyl ester and glycerin, the purified alcohol product being added to the transesterification reactor at a rate that is greater than the stoichiometric amount of alcohol required for the alkali catalyzed transesterification reaction;

separating the plurality of fatty acid alkyl esters from the glycerin in the

transesterification effluent stream in a continuous operation, wherein a first liquid phase in which the plurality of fatty acid methyl esters are concentrated and a second liquid phase in which glycerin is concentrated are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

purifying the fatty acid alkyl ester rich stream in a fatty acid alkyl ester distillation column or a fatty acid alkyl ester fractionation column and recovering the alcohol from it to produce a purified biodiesel product and a first wet alcohol stream;

purifying the glycerin rich stream and recovering the alcohol from it to produce the purified glycerin product and a second wet alcohol stream, wherein the alkali in the glycerin rich stream is reacted with an acid to produce an insoluble salt that is removed from the glycerin rich stream and thereafter filtered and rinsed with the alcohol, the pH of the glycerin rich stream being adjusted to neutral and the glycerin rich stream being further purified in a glycerin distillation column or a glycerin fractionation column and in a decolorization column; and

purifying the wet alcohol streams by removing excess water to produce a purified alcohol product, wherein the wet alcohol streams are purified in an alcohol distillation column or an alcohol fractionation column.

3. (Original.) The process of claim 2 further comprising:

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid.

4. (Original.) The process of claim 2 further comprising:

recycling at least a portion of the purified alcohol product into the transesterification

reactor for reaction with the glyceride.

5. (Currently amended.) A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

~~a step for~~ continuously conditioning the lipid feedstock to produce a conditioned lipid feedstock;

~~a step for~~ continuously measuring the concentration of the free fatty acid in the conditioned lipid feedstock by means of an in-line free fatty acid titration device that produces a signal;

~~a step for~~ continuously reacting the free fatty acid in the conditioned lipid feedstock in a glycerolysis reaction, wherein the free fatty acid in the feedstock is reacted with glycerin to produce a glyceride in response to the signal from the in-line free fatty acid titration device;

~~a step for~~ continuously reacting the glyceride in a transesterification reaction, wherein the glyceride is converted to a fatty acid methyl ester and glycerin via an alkali catalyzed;

~~a step for~~ continuously separating the fatty acid methyl ester from the glycerin to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

~~a step for~~ continuously purifying the fatty acid methyl ester rich stream and recovering the methanol from the fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet methanol stream;

~~a step for~~ continuously purifying the glycerin rich stream and recovering the methanol from the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;

~~a step for~~ continuously purifying the wet methanol streams to produce a purified

methanol product; and

~~a step for~~ recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid.

6. (Currently amended.) The process of claim 5 further ~~comprising:~~
~~——a step for comprising~~ recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.

7. (Currently amended.) A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:
~~introducing a lipid feedstock to a conditioning reactor;~~
~~——conditioning the lipid feedstock, wherein the lipid feedstock is heated, mixed and filtered to produce a conditioned lipid feedstock;~~

reacting the free fatty acid in the ~~conditioned~~ lipid feedstock in a glycerolysis reactor, wherein the free fatty acid in the feedstock is continuously reacted with a stoichiometric excess of glycerin to produce a glyceride via a glycerolysis reaction;

reacting the glyceride in a transesterification reactor, wherein the glyceride is continuously converted to a fatty acid methyl ester and glycerin via an alkali catalyzed transesterification reaction; and

separating the fatty acid methyl ester from the glycerin glycerin, ~~wherein a first liquid phase in which the fatty acid methyl ester is concentrated and a second liquid phase in which glycerin is concentrated are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;~~

~~—purifying the fatty acid methyl ester rich stream and recovering the methanol from the fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet methanol stream;~~

~~—purifying the glycerin rich stream and recovering the methanol from the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;~~

~~—purifying the wet methanol streams by removing water from them to produce a purified methanol product;~~

~~—recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid; and~~

~~—recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.~~

8. (Currently amended.) The process of claim 7 wherein the ~~introducing the lipid feedstock step further comprises:~~

~~introducing a feedstock that includes~~

~~at least one free fatty acid at a concentration in the range of about 3 percent to about 97 percent by weight;~~

~~moisture, impurities and unsaponifiable matter at a concentration up to about 5 percent by weight; and~~

~~a remainder that includes monoglycerides, diglycerides and/or triglycerides.~~

9. (Currently amended.) The process of claim ~~7~~ 10 wherein the conditioning the lipid feedstock step produces a conditioned feedstock that is a substantially uniform mixture

of liquid lipids having a temperature in the range of about 35°C to about 250°C.

10. (Currently amended.) The process of claim 9 7 wherein the ~~conditioning~~
~~the lipid feedstock~~ feedstock, prior to being reacted with the free fatty acid, is treated to render a
~~step produces a conditioned feedstock having a temperature in the range of about 45°C to about~~
65°C.

11. (Currently amended.) The process of claim 7 wherein ~~the conditioning the lipid~~
~~feedstock step produces a conditioned feedstock that is a substantially free of insoluble solids ,~~
wherein a first liquid phase in which the fatty acid methyl ester is concentrated and a second
liquid phase in which glycerin is concentrated are continuously separated to produce a fatty acid
methyl ester rich stream and a glycerin rich stream;

_____purifying the fatty acid methyl ester rich stream and recovering the methanol from the
fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet
methanol stream;

_____purifying the glycerin rich stream and recovering the methanol from the glycerin rich
stream to produce a purified glycerin product and a second wet methanol stream;

_____purifying the wet methanol streams by removing water from them to produce a purified
methanol product;

_____recycling at least a portion of the purified glycerin product into the glycerolysis reactor
for reaction with the free fatty acid; and

_____recycling at least a portion of the purified methanol product into the
transesterification reactor for reaction with the glyceride.

12. (Original.) The process of claim 7 wherein the reacting the free fatty acid step further comprises:

combining the free fatty acid with an effective amount of glycerin for an effective amount of time to facilitate the glycerolysis reaction under conditions wherein the free fatty acid and the glycerin come into substantially intimate contact.

13. (Original.) The process of claim 12 further wherein a low frequency acoustic transducer is used to mix the free fatty acid and the effective amount of glycerin.

14. (Original.) The process of claim 12 wherein the reacting the free fatty acid step further comprises:

performing the glycerolysis reaction at a temperature in the range of about 150°C to about 250°C; and

removing water from the glycerolysis reactor.

15. (Original.) The process of claim 14 wherein the water is removed as vapor through a fractionation column or a distillation column that returns condensed glycerin to the glycerolysis reactor.

16. (Original.) The process of claim 7 wherein the reacting the glyceride step further comprises:

contacting the glyceride with an effective amount of methanol and an effective amount of alkali catalyst under conditions wherein the glyceride, the effective amount of methanol and the

effective amount of alkali catalyst come into substantially intimate contact; and

wherein the effective amount of the alkali catalyst is selected from the group consisting of

an effective amount of sodium hydroxide, and

an effective amount of potassium hydroxide.

17. (Original.) The process of claim 16 wherein the reacting the glyceride step further comprises:

performing the transesterification reaction at a temperature in the range of about 20°C to about 250°C.

18. (Original.) The process of claim 17 wherein the reacting the glyceride step further comprises:

performing the transesterification reaction at a temperature in the range of about 55°C to about 65°C.

19. (Original.) The process of claim 7 wherein the reacting the glyceride step further comprises:

performing the transesterification reaction at an absolute pressure in the range of about 1 bar to about 250 bar.

20. (Original.) The process of claim 19 wherein the reacting the glyceride step further comprises:

performing the transesterification reaction at an absolute pressure of about 1 bar.

21. (Original.) The process of claim 16 further wherein a low frequency acoustic transducer is used to mix the glyceride, the effective amount of methanol and the effective amount of alkali catalyst.

22. (Original.) The process of claim 7 wherein the separating the fatty acid methyl ester from the glycerin step involves using the density difference between the first liquid phase and the second liquid phase to separate them in a continuous operation.

23. (Original.) The process of claim 7 wherein the purifying the fatty acid methyl ester rich stream step further comprises:

using a fatty acid methyl ester distillation column or a fractionation column to separate the fatty acid methyl ester rich stream into a bottoms fraction, an overhead fraction comprising primarily methanol, and a side stream fraction comprising a fatty acid methyl ester product.

24. (Original.) The process of claim 23 wherein the bottoms fraction produced by the fatty acid methyl ester distillation column or fractionation column comprises impurities, and unsaponifiable materials, unreacted monoglycerides, unreacted diglycerides, unreacted triglycerides and fatty acids.

25. (Original.) The process of claim 23 wherein the fatty acid methyl ester product produced by the fatty acid methyl ester distillation column meets ASTM specification D

6751-02.

26. (Original.) The process of claim 23 wherein the overhead fraction produced by the fatty acid methyl ester distillation column or fractionation column comprises essentially methanol.

27. (Original.) The process of claim 23 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a pressure below about 2 pounds per square inch absolute.

28. (Original.) The process of claim 27 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a pressure in the range of about 0.1 pounds per square inch absolute to about 2 pounds per square inch absolute.

29. (Original.) The process of claim 23 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a temperature in the range of about 180°C to about 280°C.

30. (Original.) The process of claim 29 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a temperature in the range of about 180°C to about 230°C.

31. (Original.) The process of claim 17 wherein the fatty acid methyl ester

distillation column or fractionation column contains a packing material.

32. (Original.) The process of claim 7 wherein the purifying the glycerin rich stream and recovering methanol step further comprises:

performing glycerin fractionation, wherein the fractions within the glycerin rich stream are separated by distillation;

performing phase separation, wherein the impurities that co-fractionate with glycerin are removed by immiscibility and differences in density; and

glycerin polishing, wherein other impurities are removed from glycerin.

33. (Original.) The process of claim 32 wherein the purifying the glycerin rich stream and recovering methanol step further comprises:

performing alkali catalyst precipitation, wherein the glycerin rich stream is reacted with a mineral acid suitable to form an insoluble salt with the alkali catalyst used in the transesterification reaction, wherein the mineral acid is selected from the group consisting of

sulfuric acid, and

phosphoric acid;

performing solids separation, wherein the insoluble salt is removed from the liquid permeate;

performing phase separation, wherein a fatty acid methyl ester rich liquid phase and a glycerin rich liquid phase are separated;

performing pH adjustment, wherein the pH of the glycerin rich stream is adjusted by adding an alkali solution;

performing glycerin fractionation, wherein the glycerin rich stream is purified by means of a glycerin distillation column and methanol is collected for further purification and reuse in the process; and

performing glycerin polishing, wherein colored impurities are removed from the glycerin.

34. (Original.) The process of claim 33 wherein the performing glycerin phase pH adjustment step is performed using ion exchange media.

35. (Original.) The process of claim 33 wherein the insoluble salt is separated using a rotary vacuum drum filter, a plate and frame press or a belt press.

36. (Original.) The process of claim 33 wherein the alkali catalyst and mineral acid used for alkali catalyst precipitation are chosen so that their reaction will produce a byproduct salt having fertilizer value; said byproduct salts are washed free of organic materials with a solvent to produce a purified salt and the purified salt is then dried and the solvent is recovered for reuse in the process.

37. (Original.) The process of claim 33 wherein the insoluble salt is washed free of organic impurities with a solvent prior to the performing solids separation step or during the performing solids separation step using filtration equipment.

38. (Original.) The process of claim 37 further comprising drying the insoluble salt in a drier under conditions wherein temperature of the drier exceeds the boiling point of the

solvent at the operating pressure of the dryer; the dryer is optionally operated under a vacuum to improve the drying; and the drier includes a condenser to recover the solvent for reuse.

39. (Original.) The process of claim 38 wherein the solvent is methanol.

40. (Original.) The process of claim 33 wherein the insoluble salt is further processed for use as a fertilizer by dissolving it in water.

41. (Original.) The process of claim 33 wherein the performing glycerin fractionation step further comprising:

distilling the neutralized crude glycerin stream to produce a bottoms material, a side stream and an overhead stream.

42. (Original.) The process of claim 33 wherein the bottoms material contains essentially waste materials; the side stream contains essentially glycerin and trace impurities; and the overhead stream contains essentially water and methanol that is recovered and recycled.

43. (Original.) The process of claim 33 wherein the glycerin distillation column is operated under a reduced pressure of below about 2 pounds per square inch absolute.

44. (Original.) The process of claim 43 wherein the glycerin distillation column contains packing material and is operated under a reduced pressure of between about 0.1 and about 2 pounds per square inch absolute.

45. (Original.) The process of claim 33 wherein the glycerin distillation column is operated at an elevated temperature between about 180°C and about 280°C.

46. (Original.) The process of claim 45 wherein the glycerin distillation column contains packing material and is operated at an elevated temperature between about 180°C and about 230°C.

47. (Original.) The process of claim 33 wherein the glycerin polishing step comprises contacting the glycerin with activated carbon at a temperature that is between about 35°C and 200°C.

48. (Original.) The process of claim 47 wherein the glycerin polishing step comprises contacting the glycerin with a packed bed of activated carbon for a contact time of less than four hours at a temperature that is between about 40°C and 100°C and wherein activated carbon fines carried through the packed bed are removed by filtration through a hydrophilic filter material.

49. (Original.) A process for production of biodiesel and glycerin comprising:
inputting to a glycerolysis reactor an effective amount of glycerin and a feedstock comprising 3 to 100 percent free fatty acids and, optionally, a fat and/or an oil;

reacting in the glycerolysis reactor the glycerin and at least a portion of the feedstock in an esterification reaction, with removal of water, to continuously produce a first intermediate

product comprising glycerides (including monoglycerides, diglycerides and/or triglycerides) and essentially no water;

performing in a transesterification reactor continuous base-catalyzed transesterification of the intermediate product to produce a second intermediate product comprising fatty acid methyl esters and glycerin;

continuously treating the second intermediate product to separate the fatty acid methyl esters from the glycerin to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

continuously purifying the fatty acid methyl ester rich stream and recovering methanol from it to produce a purified biodiesel product and a first wet methanol stream;

continuously purifying the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;

continuously purifying the wet methanol streams to produce a purified methanol product; and

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acids; and

recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glycerides.

50. (Original.) The process of claim 49 wherein the feedstock comprises an animal fat and/or a vegetable oil.

51. (Original.) The process of claim 49 wherein the effective amount of glycerin

is about two times the stoichiometric amount of fatty acids in the feedstock.

52. (Original.) The process of claim 49 wherein the reacting step is carried out at a temperature in the range of about 200°C to about 250°C, under agitation and to the extent that the first intermediate product contains no more than 0.5 percent w/w of free fatty acids.

53. (Original.) The process of claim 49 wherein the performing step comprises adding potassium methoxide to the intermediate product to facilitate base catalysis and wherein the performing step is carried out at a temperature in the range of about 40°C to about 60°C.

54. (Cancelled.)

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70. (Cancelled.)

71. (Cancelled.)

72. (Cancelled.)

73. (Cancelled.)

74. (Cancelled.)

75. (Cancelled.)

76. (Cancelled.)

77. (New.) A process of making fatty acid methyl esters from a free fatty acid feedstock, comprising:

(A.) converting the free fatty acid in the feedstock to mono-, di- and tri-glycerides by adding a glycerin product to the feedstock;

(B.) transesterifying the glycerides into fatty acid methyl esters by the addition of an alcohol to the mono-, di- and tri-glycerides; and

(C.) recovering the fatty acid methyl esters.

78. (New.) The process of Claim 77, wherein the free fatty acid in step (A) is converted to mono-, di- and tri-glycerides in the absence of a catalyst.

79. (New.) The process of Claim 77, wherein the glycerides are tranesterified with methanol in step (B.) in the presence of an alkali catalyst.

80. A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

- (A.) reacting the free fatty acid in the lipid feedstock with glycerin in the absence of a catalyst to form glycerides,
- (B.) reacting the glycerides with an alcohol to produce a transesterified fatty acid alkyl ester and glycerin; and
- (C.) separating the plurality of fatty acid alkyl esters from the glycerin.

81. (New.) The process of Claim 77, wherein prior to step (A.) the feedstock is conditioned to remove solids.

82. (New.) The process of Claim 80, wherein the free fatty acid is converted to the mono-, di- and tri-glycerides in the absence of a catalyst.

83. (New.) The process of Claim 77, wherein the glycerides are transesterified with methanol in the presence of potassium hydroxide.

84. (New.) The process of Claim 77, wherein glycerin in step (A.) is continuously added at a rate greater than the stoichiometric amount of glycerin required for glycerolysis.

85. (New.) The process of Claim 77, wherein the alcohol is added at a greater rate than the stoichiometric amount of alcohol required for the transesterification reaction.

86. (New.) The process of Claim 80, wherein in step (C.) a first liquid phase containing the plurality of fatty acid methyl esters and a second liquid phase containing glycerin are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream.

87. (New.) The process of Claim 86, wherein the fatty acid methyl ester rich stream is purified in a distillation or fractionation system.

88. (New.) The process of Claim 87, further comprising recovering a first wet alcohol stream from the fatty acid alkyl ester rich stream

89. (New.) The process of Claim 88, further comprising recovering alcohol from the glycerin rich stream and a second wet alcohol stream.

90. (New.) The process of Claim 86, further comprising reacting the glycerin rich stream with an acid to render a salt.

91. (New.) The process of Claim 90, further comprising removing, filtering and rinsing the salt with an alcohol.

92. (New.) The process of Claim 91, wherein the pH of the glycerin rich

stream is adjusted to neutral and then purified in a distillation or fractionation system and is then decolorized.

93. (New.) The process of Claim 77, wherein the effluent stream from step (A.) contains less than 0.5 percent by weight of free fatty acids and a plurality of glycerides.

94. (New.) The process of Claim 77, wherein in step (A.) glycerin is continuously added at a rate in the range of about 110 percent to about 400 percent of the stoichiometric amount of glycerin required for glycerolysis.

95. (New.) The process of Claim 80, wherein after step (A.) vapor is continuously removed to a fractionation system that returns a condensed glycerin rich stream.

96. (New.) The process of Claim 80, wherein step (A.) is conducted in at least two continuous stirred tank reactors.

97. (New.) The process of Claim 77, wherein the alcohol is methanol.

98. (New.) The process of Claim 97, wherein the at least two reactors have a combined residence time of not more than about 500 minutes.

99. (New.) The process of Claim 77, wherein in step (B.) methanol is added at a rate equal to about 200 percent of the stoichiometric amount of methanol required for

transesterification.

100. (New.) The process of Claim 80, wherein the glycerides are reacted with the alcohol in the presence of an alkali catalyst.

101. (New.) The process of Claim 80, wherein transesterification is conducted in two continuous stirred tank reactors.